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Cite 2

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PATENT SPECIFICATION

346,774

Application Date: Jan. 9, 1930. No. 864/30.
" " May 27, 1930. No. 16,357/30.
One Complete Left: Oct. 9, 1930.
Complete Accepted: April 9, 1931.

PROVISIONAL SPECIFICATION.

No. 864, A.D. 1930.

Process for the Electrolytic Manufacture of Gaseous Fluorine.

I, Arthur Carpmael, British Subject, of 24, Southampton Buildings, London, W.C. 2, do hereby declare the nature of this invention (which has been communicated to me by I. G. Farbenindustrie Aktiengesellschaft, a Joint Stock Company organised under the laws of Germany, of Frankfort-on-Main, Germany), to be as follows:

This invention relates to the manufacture of fluorine by electrolysis, principally from alkali-metal di- or poly-fluorides.

Various publications are available relating to the production of fluorine. Fluorine gas has hitherto been manufactured principally in accordance with the processes of Moissan and Argo, Mathers, Humiston and Anderson. In all these processes the electrodes were inserted from above into the electrolysing vessel and fluorspar was employed as jointing material for the fixation of the electrodes.

In accordance with the present invention the introduction of the electrodes into the electrolyte is effected from below, whereby the employment of the electrolyte itself in the solid form as the jointing material for the fixation of the electrode is rendered possible. The advantages resulting in contradistinction to the hitherto known processes signify an important technical advance.

The process is illustrated by the following example:—

The limbs of a U-shaped electrolysing vessel have two attachment tubes underneath. Through these are inserted the electrodes, known per se. For the purpose of jointing a copper plate or disc is employed, through which the electrodes penetrate. After the electrodes have been placed into the desired position both limbs of the electrolysing vessel are filled with thoroughly dried alkali metal difluoride and freed from the last remaining traces of moisture by passing through a thoroughly dried current of hot air. When this is accomplished the alkali metal difluoride is melted by an appropriate heating appliance and after all the alkali metal difluoride present in the attachment tubes above the copper plates or discs has become liquid it is caused to solidify and the copper plates or discs are removed in an appropriate manner. In order to prevent moisture penetrating through the attachment tubes these are now dipped into a bath of liquid paraffin wax or similar material.

The electrolysis can now proceed. Instead of alkali metal difluoride alkali metal polyfluoride, produced by the subsequent passing in of hydrogen fluoride into the alkali metal difluoride, can likewise be used for electrolysis.

Dated the 9th day of January, 1930.

CARPMAELS & RANSFORD,
Agents for Applicant,
24, Southampton Buildings, London,
W.C. 2.

PROVISIONAL SPECIFICATION.

No. 16,357, A.D. 1930.

Process for the Electrolytic Manufacture of Gaseous Fluorine.

I, Arthur Carpmael, British Subject, of 24, Southampton Buildings, London, W.C. 2, do hereby declare the nature of this invention (which has been communicated to me by I. G. Farbenindustrie Aktiengesellschaft, a Joint Stock Company organised under the laws of Germany, of Frankfort-on-Main, Germany), to be as follows:—

This invention relates to an improve-

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ment in or modification and further development of the process of specification No. 864/1930.

Specification No. 864/1930 describes a process and apparatus for the manufacture of fluorine by electrolysis of alkali metal di- or polyfluorides, in which the electrodes are introduced from below into the electrolysing vessel the solidified electrolyte itself being employed as jointing material.

In accordance with the present invention it may likewise be of advantage in the case of complicated apparatus to introduce the electrodes from above or from the sides with the result that the same can be more readily replaced. In this arrangement also solidified electrolyte, that is to say solid alkali metal di- or polyfluoride, is employed as the jointing material.

The following example illustrates the invention:—

The electrolysing vessel is provided at its upper openings with attachments, the rims of which are given the form of a cup. Each electrode is provided with a cap. The cups are filled with the electrolyte and the caps of the respective electrodes are dipped into the electrolyte, which is then allowed to solidify. The execution of the electrolysis is effected as indicated in specification No. 864/30.

Dated the 27th day of May, 1930.

CARPMAELS & RANSFORD,
Agents for Applicant,
24, Southampton Buildings, London,
W.C. 2.

COMPLETE SPECIFICATION.

Process for the Electrolytic Manufacture of Gaseous Fluorine.

I, Arthur Carpmael, British Subject, of 24, Southampton Buildings, London, W.C. 2, do hereby declare the nature of this invention (which has been communicated to me by I. G. Farbenindustrie Aktiengesellschaft, a Joint Stock Company organised under the laws of Germany, of Frankfort-on-Main, Germany), and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the manufacture of fluorine by electrolysis from acid fluorides of alkali-metals including those containing more than one molecular proportion of hydrogen fluoride.

Various publications are available relating to the production of fluorine. Fluorine has hitherto been manufactured principally in accordance with the processes of Moissan and Argo, Mathers, Humiston and Anderson. In all these processes the electrodes were inserted from above into the electrolysing vessel and fluorspar was employed as jointing material for the fixation of the electrodes.

In accordance with the present invention the electrolyte itself in the solid form is employed as the jointing and insulating material for the fixation of the electrodes. The advantages resulting in contradistinction to the hitherto known processes signify an important technical advance. The electrodes are introduced from below upwardly into the electrolyte or from above or from the sides by means of cups or similar acting devices.

The accompanying drawing illustrates schematically in section two forms of construction of an apparatus which may be employed in the present process.

In figure 1 the limbs of a U-shaped electrolysing vessel 1 have two attachment tubes underneath. Through these are inserted the electrodes 3, known per se. For the purpose of jointing a plate or disc 6 of a metal such as copper or magnesium may be employed, through which the electrodes penetrate. Instead of metal plates, plates of an insulating material such as asbestos slate can likewise be used. After the electrodes have been placed into the desired position both limbs of the electrolysing vessel are filled with thoroughly dried alkali metal acid fluoride (bifluoride) and freed from the last remaining traces of moisture by passing through a thoroughly dried current of hot air. When this is accomplished the alkali metal acid fluoride is melted by an appropriate heating appliance and after all the alkali metal acid fluoride present in the attachment tubes above the metal or insulating material plates or discs has become liquid it is caused to solidify at 5 by means of the cooling jackets shown and the metal plates or discs are removed in an appropriate manner while the plates or discs made from insulating materials need not be removed. In order to prevent moisture from penetrating through the attachment tubes these are now dipped into a bath of liquid paraffin wax or similar material.

The electrolysis can now proceed. During the electrolysis hydrofluoric acid freed from water is added through 4 in the same measure as fluorine and hydrogen are evolved from the electrolyte. In this way the composition of the electrolyte is maintained constant so that a continuous preparation of fluorine is guaranteed. During the electrolysis some hydrogen fluoride tends to escape by evaporation in consequence of the high temperature of the melt. This escape can be prevented by attaching a cooler 2 to the tubes through which the evolved fluorine is led. At this point the tubes are advantageously expanded like a steam-dome in order to prevent stoppage by any of the solid substance separated there. The cooler acts as a reflux condenser which prevents any evaporation of hydrogen fluoride. At the same time a greater partial pressure of hydrogen fluoride above the melt is obtained so that by this means also the evaporation of hydrogen fluoride is restrained. Instead of alkali metal bifluoride an alkali metal acid fluoride containing more than one molecular proportion of hydrogen fluoride, produced by the subsequent passing of hydrogen fluoride into the alkali metal bifluoride, can likewise be used for the electrolysis.

It may likewise be of advantage in the case of complicated apparatus to introduce the electrodes from the sides or from above as in figure 2 in which 7 shows a cup filled with solidified electrolyte 5 and the electrode has an inverted smaller cup 8 held by the solid electrolyte. In this arrangement also solidified electrolyte, that is to say solid alkali metal acid fluoride, is accordingly employed as the jointing material. Instead of a U-shaped electrolysing vessel as in figure 1 any other appropriate electrolysing vessel may be employed as for example an electrolysing vessel of cylindrical or prismatic shape, containing diaphragms 9 (figure 2) which allow the passage of the electrolyte from the cathode to the anode chamber but prevent the passage of gases from one chamber to the other.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. In the manufacture of fluorine by electrolysis from acid fluorides of alkali metals including those containing more than one molecular proportion of hydrogen fluoride, the application of the solid electrolyte as jointing and insulating material for the leading-in of the electrodes.

2. Process as claimed in claim 1, in which the electrodes are introduced from below upwardly into the electrolyte or from above or from the sides by means of cups or similarly acting devices.

3. Process as claimed in claim 1 in which in order to render the process continuous hydrofluoric acid is added to the electrolyte in the same quantity as fluorine and hydrogen are developed in the electrolysis.

4. The improved process for the manufacture of gaseous fluorine substantially as described with reference to the accompanying drawing.

Dated the 9th day of October, 1930.

CARPMAELS & RANSFORD,
Agents for Applicant,
24, Southampton Buildings, London,
W.C. 2.

Redhill: Printed for His Majesty's Stationery Office, by Love & Malcomson, Ltd.—1931.

[This Drawing is a reproduction of the Original on a reduced scale]

Fig. 1.

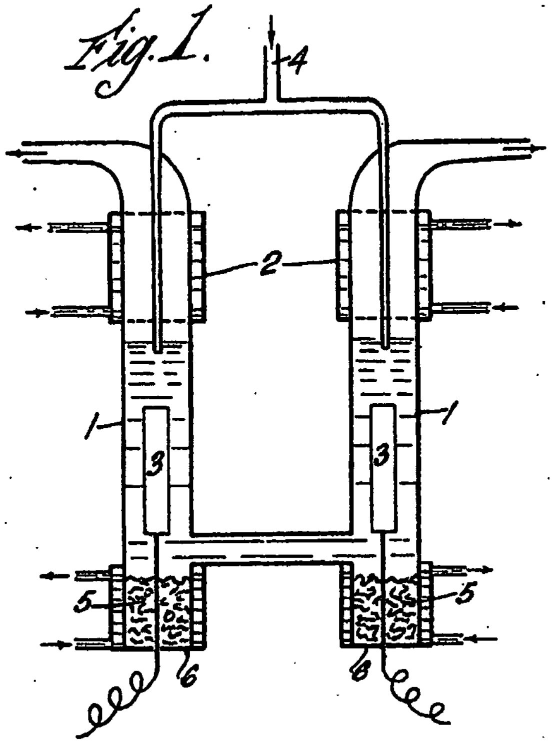
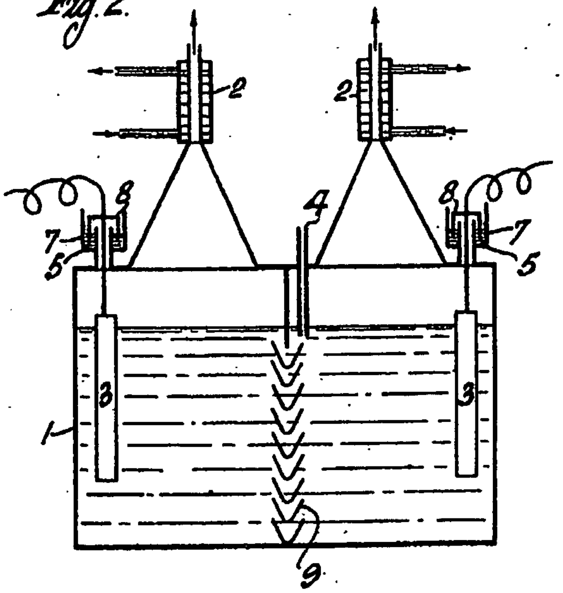


Fig. 2.



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